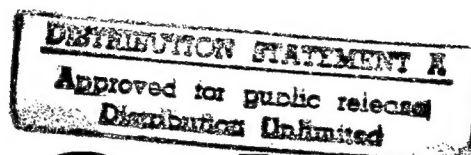




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Science & Technology

***Japan
Fine Ceramics: Past, Present, Future***

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Science & Technology

Japan

Fine Ceramics: Past, Present, Future

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08 JUNE 1990

ADVANCED MATERIALS

Fine Ceramics: Past, Present, Future

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Fine Ceramics: Past, Present, Future

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KENKYU NO AYUMI in Japanese 21 Nov 88 pp 1-926

[Foreword, Editor's Note, Table of Contents, pp 3-11, 15-16, 175-176, 921-926 of the book "The Course of Fine Ceramics R&D, A Next Generation's Industrial Key Technology R&D," The Fine Ceramics Technical Research Association, Tokyo, 1988, 926 pages]

[Excerpts]Foreword

Hideaki Yamashita, chairman of the Board of Directors,
Fine Ceramics Technical Research Association

The next generation's industrial key technology R&D system (the System), as a creative measure by the Agency of Industrial Science and Technology (AIST), MITI, for establishing the foundation for Japan's future industries and for nurturing independent technologies, is a unique system that strictly sets its research objectives on technical development in order to secure the foundation for common, high technological standards. Both its concept and timing could not have been more appropriate, and speaking from the industrial community, I was compelled to sincerely express my respect and support to the System. The "fine ceramics R&D" project, being a part of the System and belonging to the field of new materials, has targeted the establishment of industrial structural materials that would open a new horizon which could not have been reached with existing materials, in particular, with their heat resistance, corrosion resistance and abrasion resistance. It is worth special mention that, since the project inauguration, closer-than-ever cooperation has been maintained between the industry, government and universities, and integrated efforts have been demonstrated. As a result, through the first and the second phases in the past, not only the world's most reliable silicon nitride and silicon carbide materials have been successfully developed, but also it gives me great pleasure to have witnessed internationally respectable accomplishments in the development of individual related technologies including raw powder material technology, mold-sintering and fabrication bonding process technology, evaluation technology and application technology. While these achievements were made, I can recognize the significantly positive aspects of the next generation symposia, academic societies' meetings and academic journal reports from abroad, which stimulated research concerning the above-mentioned technologies conducted throughout Japan and propagated vitalization in both competition and cooperation with respect to the related business activities.

The fine ceramics R&D was further expanded to include the search for high-toughness materials. Currently, this effort is being continued based on the plan for the next five years in an attempt to comprehensively demonstrate new materials in the coal gasification cogeneration ceramic turbine part elemental technology. However, because of the public's keen interest in the above-mentioned accomplishments of the first and second

phases, it was planned to compile the entire results in one book for publication and to request appraisals and comments on it. Fortunately, the plan was blessed by the public's understanding of the meaning of this unique national project, and it would give me an unexpected pleasure if the book can contribute to further enrichment and progress of R&D for achieving the goal.

I would like to express my most sincere appreciation for the policies of MITI and AIST, MITI, which paved a road for the drastic progress of Japan's industrial technology by creating the System, and for the guidance by the academic community. I would also like to express my gratitude to government research organizations, national universities and many members of the Association for their never-failing active cooperation in their execution of research and in this publication plan.

November 1988

Foreword

Kozo Iizuka, director-general, AIST, MITI

With the 21st century immediately upon us, our hopes are soaring higher and higher for technology development in such advanced fields as advanced materials, electronics and biotechnology. It is mandatory for Japan to aggressively challenge R&D for new technologies, in order to overcome its lack of resources and territories, raise the world's technological levels through technical revolution, and positively contribute to the international society.

With this understanding, AIST has pursued "the System" for the purpose of establishing the next generation's industries, including the aerospace, information processing and biotechnological industries, and of developing revolutionary key technologies that are indispensable for the sophistication of existing diverse industries. Today, a total of 14 R&D projects are carried out under this System.

One of the projects, "fine ceramics R&D," has been undertaken as a 12-year, three-phase plan, from FY81 through FY92, for the purpose of developing fine ceramics having excellent properties, such as high strength at high temperatures, high corrosion resistance and high abrasion resistance, that have never been observed before.

The first two phases had already been completed by FY87. During this period, not only the world's highest level of achievements have been made in the high-temperature strength and antioxidation property, but also significant achievements have been made in the area of scientific elucidation of mechanisms in the ceramic field which previously, comparatively speaking, lacked theoretical substantiations. These achievements, drawing international attention, have been reported through many opportunities, including academic societies' meetings and symposia. With the completion of the second phase of this project, I am extremely happy to

see that the Fine Ceramics Technical Research Association (Association) is soon to publish the book, "The Course of R&D Concerning Fine Ceramics, the Next Generation's Industrial Key Technology," by gathering all the previous achievements. I firmly believe that, through this book, everyone connected with the field will be able to systematically and comprehensively understand what this project has accomplished.

I sincerely hope that this book will be the guidelines for future R&D both in Japan and abroad, and I pray that this project will continue to create many more achievements through the efforts of the people concerned.

November 1988

Foreword

Shinroku Saito, professor emeritus, Tokyo Institute of Technology

An enormous amount of materials was collected. Although the book is supposed to be a limited edition, if it can be sent to those concerned and can arouse public opinion among informed people of the world, I will be undeservedly happier for what little I have done for fine ceramics R&D under the System.

As you well know, many projects under the System are concerned with raw materials. Ceramics, that were previously regarded as a pronoun for brittle things, have been in the limelight as high-temperature, high-strength materials with completely new functions. It is noteworthy in the history of technology that the industry, government and universities together began R&D to develop structural materials for heat engines, including gas turbines, from ceramics. Needless to say, ceramics, breaking out of its previously commonly accepted idea associated with rice bowls and tiles, already entered the markets of electrical insulators, oxide magnets, powerful inductors and varistors. Recently, ceramics have become popular as a possible answer to diverse demands from optoelectronics to superconductivity. However, it has been a tremendous challenge to improve ceramics' strength and toughness, regardless of use temperatures, high or low. For those who had been engaged in the traditional earthenware and pottery industries, the improvement effort was a dream, or in some cases, even a taboo. Although a clue to the solution to this problem began to appear on the market in the form of alumina chips for cutting tools, the U.S. cermet R&D and its application to high temperatures had to be hopelessly abandoned prior to the AGT plan.

Thus, the application for gas turbines, tried before the next generation project came into the picture, had been finished without arousing public opinion. When the same attempt was planned, upon reflection of the past, as a challenge for the next generation, there was deep concern still on the part of people who planned and those around them. Frankly speaking, has the concern completely gone? Although it is rather rude, and it seems as if I do not know my own place to answer the question in

the beginning of the book, the only technology in this project that has blossomed has been the application for turbo-chargers with their temperature environment not exceeding 1,000°C. Although some products have been developed to withstand higher environmental conditions at 1,400°C, the temperature target has been gradually raised up to the current 1,500°C. Paradoxically, the consensus of public opinion says that nothing other than ceramics will be able to satisfy the conditions, and therefore, a burden has been placed on the project.

At the same time, the evaluation and test methods for brittle materials have advanced almost out of recognition by repeating trials and errors. The bonding technology and other processing technologies have also been developed sufficiently to meet the market demands. And, because of all the progress, we want to present this enormous accumulation of data to you with questions. As the chairman of the committee in charge of gathering and organizing the data, as requested by the Industrial Technology Council of MITI at the time of the inauguration of the next generation project, I feel particularly fortunate to have been placed in the center of the activity for discovering solutions one after another for old, yet new, brittleness problems of ceramics. At the same time, with these massive data in front of myself, and reflecting the past, I would like to express my sincere appreciation to those who worked hard to produce the data. Hoping that they will continue to vigorously challenge yet-to-be-solved unknowns, I would like to ask again MITI, private companies and universities for continued cooperation on this matter.

November 1988

Editor's Note

The Secretariat, the Fine Ceramics Technical Research Association

Presented in this book are accomplishments in the past 7 years of the "Fine Ceramics R&D" project (including a portion of the original project that has since been renamed and separated from this project due to the introduction of the special account budget for the electric power source development promotion measures), one of the projects under the System, which was inaugurated in October 1981 as a national research project by AIST, MITI. The description and editing were done by researchers at government research organizations and private industry's research organizations, who divided research work among themselves, as well as by the secretariat of the Association. The specific details of all R&D projects are described in the more than approximately 1,000-page annual research reports that have been submitted each year to AIST. Upon closing of the second phase as defined in the basic R&D program, key points were summarized for each small individual project. In the opening chapter of this book, the comprehensive program and the approach outlines of the overall research projects are introduced, and then the descriptive contents and results of each more specific research

project are successively given in the form of an either academic or technical report. After reviewing the annual reports, some sections were completely rewritten based on chronological data spanning several years. However, for the most part, the editing was done with emphasis on the original graphs and tables taken from the annual reports, therefore, the individual report formats were not specifically standardized but were left up to the discretion of the organization in charge. In some cases, as research projects have proceeded over a long period of time, research policies were revised in consideration of objective situation changes, experimental data were improved; and because of the transfer of researchers directly involved in R&D projects, continuous thoughts and logic forms were difficult to find. In these cases, the editors' policies were to describe exactly what had taken place. The book was written all in Japanese. However, in order to serve the interests of overseas readers as much as possible, an English abstract was inserted at the beginning of each report and an effort was made to use English captions wherever possible for the explanation of graphs and tables, although there were many situations where the English translation was intentionally omitted when a judgment had been made that any English translation effort would rather obscure the meaning of an original report. The editor would like to ask the readers' understanding for the lack of uniformity in this respect. Furthermore, the readers should be prewarned that some reports only referred to the objectives of projects and omitted in part recent unpublished experimental results in order to keep researchers' proprietorships. Finally, the plan is under way to follow up on these unpublished results under one of the discussion topics at the open-to-public symposium scheduled for the fall of 1991.

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I. Preface

1. Next Generation's Industrial Key Technology R&D System

In the early 1980's, the Industrial Structure Council, MITI, published the so-called "MITI Policy Outlook for the 80's" (Mach 1980) to show the guidelines for resource-poor Japan to overcome its fragility. Accordingly, AIST, MITI, established the System to support in part the policy to promote the guidelines and to advocate for Japan to become a technological nation, that will relieve itself from the dependence on imported overseas

technologies by developing its own technologies. The primary objective of this System was to eradicate a key technology gap of allegedly 5 to 10 years between Japan and the West by promoting the development of "key technologies" behind coordinated efforts by the industry, government and universities. The "key technologies" will not only be indispensable for the establishment of the "next generation's industries," including the aerospace, information processing, new energy development and biotechnological industries, which are expected to significantly advance in the 1990's, but also be expected to contribute to the sophistication of existing diverse industries. Advanced materials, biotechnology and new functional devices were selected to be the research target fields for the System, based on the following three requirements for a key technology that needs to be developed.

1. Extremely innovative and having propagative effects over a wide range.
2. Require as long a R&D period as approximately 10 years and a large development fund, and have a high risk factor in R&D.
3. Require urgency in development initiation, because aggressive R&D was already started in other advanced nations.

Further, 12 research topics were established to these three technology fields, and in October 1981, research activities commenced. Since then, full 7 years have passed. After the commencement, superconductivity was added as the fourth technology and some research topics were terminated. Therefore, a total of 14 topics are still on the active list as of the beginning of FY88.

The scope of R&D according to the System was to pursue fundamental research topics which would eventually support industrial technologies in the budding stage to become fruit-bearing saplings. That is to say, R&D activities were to begin when it became clear that a theoretically or experimentally innovative industrial technology could be practically applied (the budding stage), and the activities were to continue until the prospect of the technology becoming an industrial technology was clear (the sapling stage) with contributions being made for technology development between the two stages. The following principles were established for carrying out R&D activities.

1. The underlying principle shall be to rely on the cooperation among three parties of the industry, universities and the government (government research institutes and laboratories). Consignments shall be given to private corporations to take advantage of the industrial community's capabilities. Government research institutes and laboratories shall conduct R&D activities to make use of their past records. In addition, depending on the nature of research specifics, cooperation shall be sought from universities.

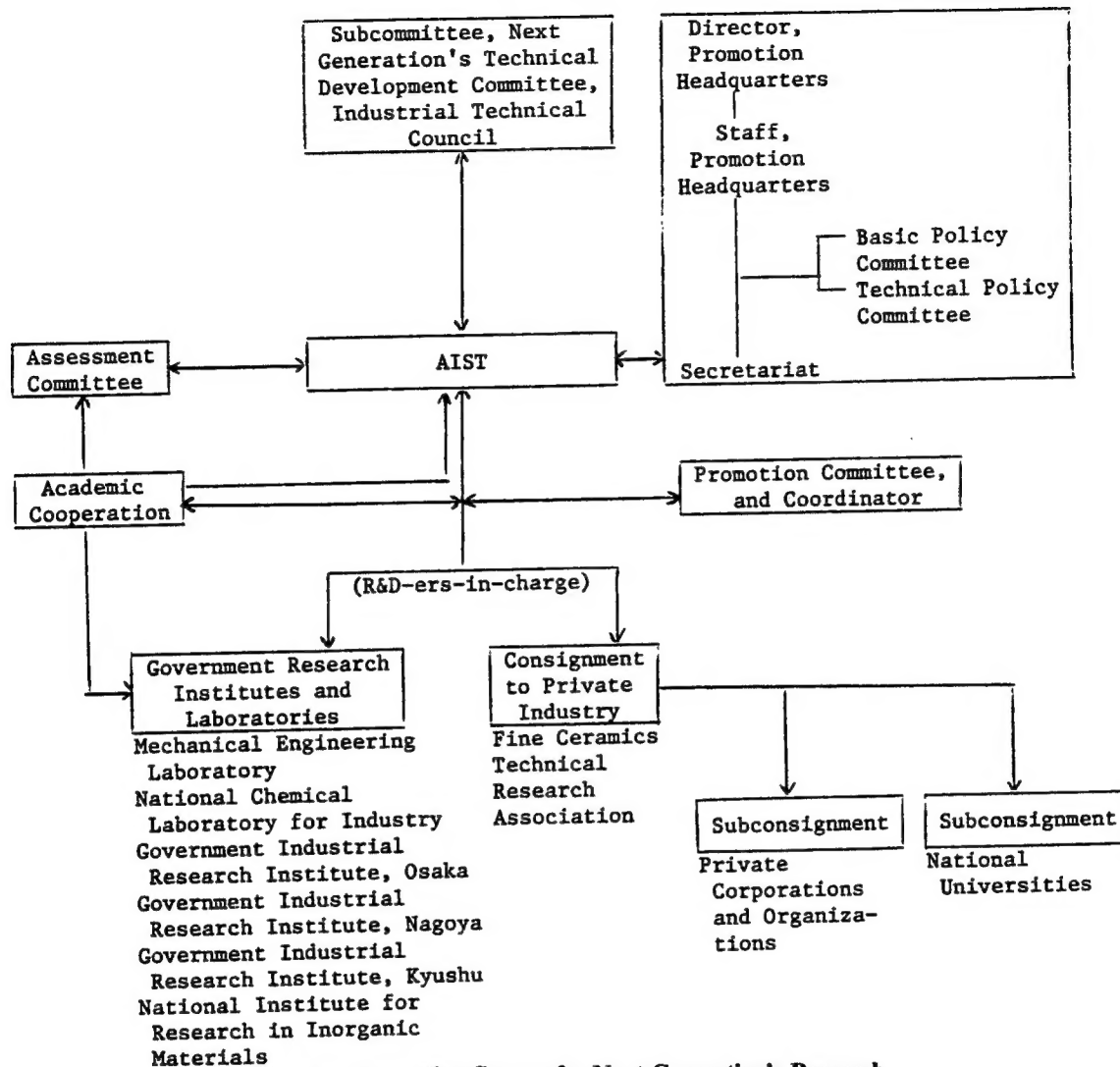


Figure 1. Promotion System for Next Generation's Research

2. In order to efficiently manage R&D activities, the parallel development technique shall be adopted, in principle, to simultaneously pursue more than one R&D method. The approximately 10-year overall plan shall be divided into three phases with each phase spanning several years, a definite target shall be set up for each phase (the phase-wise goal-setting technique), and the Assessment Committee of experts shall evaluate R&D activity status and results for each phase.

3. The below-listed organizations shall be created and each organization shall function within the system of R&D execution, as shown in Figure 1.

(1) The next generation's industrial key technology R&D promotion headquarters, the coordinating office for MITI to promote this System in connection with various industrial policies.

(2) The Industrial Technical Council, that deliberates R&D basic plans which are the basis for R&D activities over an approximately 10-year period.

(3) The Promotion Committee, that coordinates R&D activities carried out at government research institutes and laboratories and private corporations on consignment, and holds discussions on bench-level research.

(4) R&D Coordinator, who gives long-term R&D guidance and directions.

(5) The Assessment Committee, which evaluates R&D at the end of each phase.

The Promotion Committee, with the coordinator as its chairperson, consists of AIST (Office of the Next Generation's Industrial Technology Planning Officer), which is the R&D consigner, technical personnel in charge at a

consignee organization, and outside experts from the industry, government and universities. The committee's mission is to thoroughly examine R&D plans (particularly, objectives and approaches) and R&D progress status, and to give guidelines concerning the manner of R&D pursuit. The Assessment Committee, composed of only outside experts and people of learning and experience, is supposed to assist R&D promotion by submitting assessment reports to AIST. Mr Hiroshi Okuda, D.Sc. in Engineering, appointed the Coordinator, is in charge of "Fine Ceramics R&D" topics. Dr Okuda was once the chief, the Fifth Department, the Government Industrial Research Institute, Nagoya, AIST, at the time of the inauguration of the System, and is now the managing director and the Research Institute director of Fine Ceramics Center Corp.

What the government foresees in terms of the effects of R&D by this System is the blooming of revolutionary technologies that can make sweeping changes in the industry as well as the citizen's life after the 1990's. Furthermore, the government earnestly hopes that the inaugurated System will eventually be regarded to be the precursor for innovations of such a large scale as going down in history and to be the starter for enriching fundamental research concerning industrial technologies. It is further hoped that the System will play the role of a leader in building a technological nation that holds solution keys for Japan's economic society to find its way out of all kinds of future difficulties and to successfully execute its international responsibilities.

2. Fine Ceramics R&D

In FY79, prior to the System's inauguration, AIST consigned to the Japan Industrial Technology Association, a corporation, a technical assessment survey. The survey was actually undertaken by the fine ceramics technology assessment committee composed of people of knowledge and experience. This was the beginning of the research topic selection and the actual research execution concerning the ceramic material segment as a part of new material field research set forth by the System. This survey thoroughly examined and grasped the then current status and problems of fine ceramic materials with thermal and mechanical functions and related technologies. The survey also selected important technical development topics for promoting future technology development, and made a proposal on the basis of the result of analyzing future technological predictions and impacts to be made on various fields.¹ The most basic thought in this proposal was to point out the greater need for software technology development that will be the common basis than the need for individual hardware technology development. In conclusion, the following three topics were selected as urgent development topics through 1990.

(1) Assessment technology for fine ceramics as mechanical and thermal industrial materials [assessment technology]

(2) Design technology for mechanical parts to take advantage of ceramics characteristics [design technology]

(3) Production and fabrication technology to economically produce complex-shaped, precision-shaped parts [production/fabrication technology]

In addition, potential problems anticipated in each of these technologies and research topics to try to solve these problems were compiled and specifically proposed as follows.

(1) Assessment Technology

Establishment and standardization of material assessment methods;
Establishment of non-destructive inspection technology to detect microflaws;
Establishment of test methods for mechanical performance and product life assurances;
Systematization of data collection and application methods.

(2) Design Technology

Establishment of design methods that take advantage of ceramic's characteristics;
Establishment of ceramics fracture dynamics;
Execution of demonstration tests.

(3) Production/Fabrication Technology

Establishment of industrial-scale precision fabrication technology;
Establishment of forming and sintering methods for large, complex shapes;
Establishment of industrial-scale monolithic sintering method for materials difficult to sinter;
Development of large high-pressure shape-forming machines;
Establishment of basic technology for powder engineering;
Development of various bonding technologies;
Improvement of high-temperature, vacuum and high-pressure technologies;
Establishment of industrial production method for homogeneous raw materials;
Development of fabrication machines exclusively for ceramics.

Attached to this proposal, the problem of the shortage of personnel, i.e., who would be responsible for rapid progress of fine ceramics was raised. To cope with the problem, it was proposed to urgently make plans to: upgrade the research system based on cooperation with non-ceramics field researchers and engineers; enable the display of combined forces by narrowing gaps between the seed side (idea origination side) and the need side (application side); make preparations for establishing a national project; enrich cooperative systems between the industry and universities; increase R&D outlays allocated to universities and government research organizations; and subsidize private corporations.

The fine ceramics R&D basic plan under the System was essentially based on the thoughts and proposals of the above survey report. Unlike previous national projects sponsored by AIST, the fine ceramics R&D project is remarkably unique in that its research topics aims at the development of merely material technologies and not at specific devices or systems. Fine ceramics are highly regarded to be used for electronic and biological materials. However, this project will target exclusively machine part materials with high strength, high corrosion resistance and high wear resistance, as well as their secure positions as industrial materials. Therefore, it is demanded to thoroughly examine the ways to improve ceramics' fracture strength, the lack of which is the reason for not having been able to realize these goals today, from the standpoints of the production and application methods.

Ceramics are essentially a non-uniform solid, and they come under a field of material science in which knowledge has been least organized. In particular, production technologies for ceramics have traditionally depended on know-how's based on experience. The measurements of physical properties and the conditions for the measurements have been expedient and diverse. Products have been preferentially selected for certain applications by empirical judgment of the suitabilities. It was indeed the true status of the related industrial community at the time of this project planning that objective product quality standards could not be established. Realizing that the status must be urgently improved, the systematic accumulation of experimental data and the development of theoretical examinations were demanded. Thus, the objectives in the production technology area were to clarify the cause-effect relationships existing between the physical properties of ceramics raw material powders, the conditions for each unit production process, and the microstructure and performance of ceramics; and, based on these relationships, to establish production technologies that can produce uniform and surface flaw-free products with excellent reproducibility, to minimize the fluctuation of product performance, in particular fracture strength. The objectives in the application technology area were to accurately determine and theoretically analyze ceramics' fractures under diverse stress load conditions, and to establish, based on the theoretical fracture analyses, the unique ceramic part design standards. It was judged not only possible but also more meaningful to achieve these objectives by establishing a national research project than by having singular corporations and research organizations carry out separate projects from the standpoints of experimental scale, experimental volume, and technical information transmission.

Initially, the research plan was expected to cover a 10-year period starting in FY81. The period was divided into three phases and an interim progress goal was established for each phase. On the basis of evaluations by the Assessment Committee, AIST was to decide the R&D plan direction for each year. The first phase was for three years up to FY83, and the second phase was for four years up to FY87, with the remainder being the third phase. In FY86,

because the special account budget for electrical resource development promotion measures was introduced, the project was divided into three, i.e., "Development of elemental technologies for coal gasification ceramic turbines" (under the charge of the Association), "Development of elemental evaluation technologies for coal gasification ceramic gas turbines" (under the charge of government research institutes under the jurisdiction of AIST), and "Fine ceramics R&D" (under the charge of the National Institute for Research in Inorganic Materials, the Science and Technology Agency). As a result, the development of elemental technologies concerning ceramic parts suitable for gas turbines for the coal gasification cogeneration system was designated as the demonstration target for the fine ceramics R&D project. Therefore, new research topics were added as described in another chapter, and in FY88, the research project period was extended for two more years to carry out R&D on consolidation technology in FY91 and FY92. The overall plan after the revision is illustrated in Figure 2.

During the first and second phases, the specific research topic activities were carried out independently by organizations in charge. However, toward the end of the second phase, in order to clarify the relationships among the physical properties of raw material powders, their sinterability and the resulting ceramics' characteristics, the research topics concerning the raw material synthesis technology and the forming and sintering technology were carried out jointly by corporations in charge, and with the leadership of the Government Industrial Research Institute, Nagoya. A total of six government research institutes participated in R&D during the first and second phases; they were the National Engineering Laboratory, the National Chemical Laboratory for Industry, the Government Industrial Research Institute, Osaka, the Government Industrial Research Institute, Nagoya, and the Government Industrial Research Institute, Kyushu, under the jurisdiction of AIST, and the National Institute for Research in Inorganic Materials of the Science and Technology Agency (STA). The following 15 private corporations organized the Association and received R&D consignment fees: Asahi Glass Co., Ltd., Ishikawajima-Harima Heavy Industries Co., Ltd., Inoue Japax Research Co., Ltd., Kyocera Corp., Kurosaki Refractories Co., Ltd., Kobe Steel, Ltd., Shinagawa Refractories Co., Ltd., Showa Denko K.K., Sumitomo Electric Industries, Ltd., Denki Kagaku Kogyo K.K., Toshiba Corp., Toyota Motor Corp., Toyoda Machine Works, Ltd., NGK Insulators, Ltd., and NGK Spark Plug Co., Ltd. In addition, a portion of the raw material synthesis technology R&D assignment was sub-consigned to Toshiba Ceramics Co., Ltd., by the Association. Furthermore, the basic research topics concerning the formability and sinterability of raw material powder, the relationship between fabrication and material strength and the adhesion technology, were successively subconsigned to three national universities for two years each (overlapped at one time) and reports were received from the universities. (Still continuing) Although, in FY87, a portion of the added research topics was subconsigned to

	81	82	83	84	85	86	87	88	89	90	91	92
1. Material Technology												
(1) Basic Process Technology												
(2) Raw Material Synthesis Technology												
(3) Forming & Sintering Technology												
1) Clarification of Molding and Sintering Mechanisms												
2) Materials by Application Field (Monolithic) (High-strength, high corrosion resistance and high precision wear resistance)												
3) Improved Toughness Materials												
(4) Fabrication and Bonding Technology												
1) Clarification of Fabrication Mechanisms												
2) Development and Application of Fabrication Devices												
3) Adhesion Technology												
4) Surface Reinforcing Technology												
2. Evaluation Technology												
(1) Property Evaluation Technology												
1) Raw Material Powders												
2) Ceramics												
(2) Non-Destructive Testing Technology												
(3) Assurance Testing Technology												
1) Clarification of Fracture Mechanisms												
2) Product Life Prediction Technology												
3. Application Technology												
(1) Design Technology												
1) Structural Analysis, Design Standards												
2) Fracture Factor Analysis (Composite stress, shock, fatigue, corrosion and thermal fatigue)												
(2) Model Evaluation Tests												
1) Primary Models												
2) Models for Gas Turbine Part Elements												
4. Consolidation Technology												

--- Survey Research

Figure 2. Overall R&D Plan Time Table

Japan Steel Corp., details of the research activities are outside the scope of this book. In FY88, Japan Steel Corp. joined the Association.

The ceramic materials targeted for development during the first and second phases were silicon nitride and silicon carbide. They were already proven to have practical use as a machine part structural material, were of a dense texture (monolithic) which is regarded to be most promising today and in the future, and were strength-wise the most reliable in the world. In consideration of

characteristic differences in use environments, the performance target values were established, as shown in Table 1, for both ceramics in three categories of high-strength materials, high-corrosion resistance materials and high-precision wear resistance materials. It should be noted in this table that the target Weibull constant values, the indices for reliability, are set high and that all the performance values are to be obtained by testing materials after they have been kept in an atmospheric environment for 1,000 hours at the same high temperature as that of actual uses. In addition, it is specified that

the above-established performance values are to be met in the first phase with ceramic test pieces, in the second phase with ceramic models of simple shapes corresponding to their practical use purposes, and in the third phase with ceramic models of near-real, complex shapes and dimensions. From the standpoint of the overall project including related technology development, the first phase can be termed the period of basic research concerning raw material

synthesis and production process; the second phase can be termed the period of research concerning the development and improvement of various elemental key technologies; and the third phase and on can be termed the period of the integration and demonstration of all developed technologies. The actual fiscal R&D outlays through the final fiscal of the second phase, including the development expenses paid to government research organizations and the consignment fees paid to the Association, are shown in Figure 3.

Table 1. Material Performance Targets in First and Second Phases

Application Category	Target Values
High-Strength Materials	(1) After being kept at a temperature higher than 1,200°C for 1,000 hours in the atmosphere, the materials must meet the following performance values under the same conditions:
	1) Reliability (Weibull constant) m: more than 20
	2) Strength (average tensile strength) σ : greater than 30 kgf/mm ²
	(2) When subjected to 1,000-hour creep tests under an atmospheric, 1,200°C high-temperature environment, the materials must meet the following performance value:
	Durability (creep strength) σ : greater than 10 kgf/mm ²
High-Corrosion Resistance Materials	After being kept at a temperature higher than 1,300°C for 1,000 hours in the atmosphere, the materials must meet the following performance values:
	1) Reliability (Weibull constant) m: more than 20
	2) Corrosion resistance (oxidation weight gain) less than 1 mg/cm ²
	3) Strength (average tensile strength) σ : greater than 20 kgf/mm ²
High-Precision Wear Resistance Materials	(1) After being kept in an atmospheric 800°C-environment for 1,000 hours, the materials must meet the following performance values in the same environment:
	1) Reliability (Weibull constant) m: more than 22
	2) Strength (average tensile strength) σ : greater than 50 kgf/mm ²
	(2) The materials must meet the following performance values when tested at room temperature:
	1) Wear resistance (specific wear rate): less than 10 ⁻⁸ mm ³ /kg-mm
	2) Surface texture (surface smoothness) R _{max} : less than 2 μ m

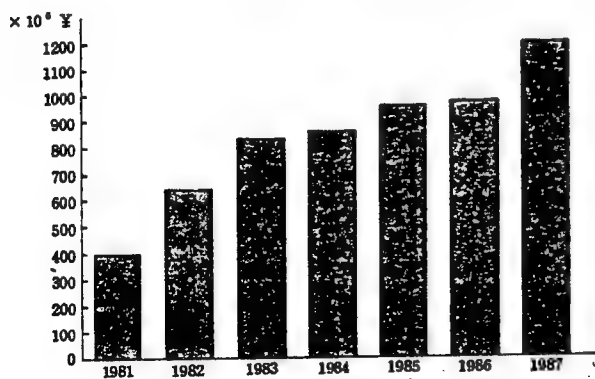


Figure 3. Fiscal R&D Outlays

As shown in Figure 2, as far as the specific research topics were concerned, the entire project was divided into three divisions of production process technology, evaluation (measurement) technology and application technology. In principle, close communications were to be maintained among these divisions in carrying out research topic activities. Because the topics concerning raw material powder and ceramic development were the starting point, the

master project began in FY81 with only the basic research topics for raw material synthesis and mold-sintering technology and the basic technology research topics for evaluation and measurement technologies and process development by government research organizations. Therefore, the close evaluation for the first phase covered only these research topics. During the course of the first phase, additional research topics were gradually initiated: they included the fracture strength measurements under various stress conditions and the model test technology, both of which were under the application technology division, and the fabrication technology and the non-destructive test technology under the production process technology division. In the second phase, more specific research topics of adhesion technology, assurance technology, structural analysis and design standards were started. By the middle of the second phase, all the technological elements, which had originally been planned, were actively pursued side by side for the first time. The line-up of these research topics and the organizations in charge, as of that time, are shown in Table 2. Since the beginning of the third phase, early-initiated topics have gradually been completed, and ultimately the integrated demonstration and evaluation of developed technologies are scheduled to be done by subjecting them to various tests for the ceramic gas turbine part elemental technologies.

Table 2. Second Phase Research Topics and Research Organizations-in-Charge

1. Process Technology	
(1) Process Development Basic Technology	
1) Process Basic Technology	Government Industrial Research Institute, Nagoya, AIST
2) Explosion Treatment Forming/Sintering Technology	National Chemical Laboratory for Industry, AIST
(2) Raw Material Powder Synthesis Technology	
1) Silicon Nitride	Fine Ceramics Technical Research Association
2) Silicon Carbide	Fine Ceramics Technical Research Association
(3) Forming/Sintering Technology	
1) High-Strength Material	Fine Ceramics Technical Research Association
2) High-Corrosion Resistance Material	Fine Ceramics Technical Research Association
3) High-Precision Wear Resistant Material	Fine Ceramics Research Association
(4) Fabrication/Bonding Technology	
1) Fabrication Technology	Mechanical Engineering Laboratory, AIST
	Fine Ceramics Technical Research Association
2) Adhesion Technology	Fine Ceramics Technical Research Association
2. Evaluation Technology	
(1) Characteristic Evaluation Technology	
1) Raw Material Powder	Government Industrial Research Institute, Nagoya, AIST
2) Ceramic Material	Government Industrial Research Institute, Nagoya, AIST
	Government Industrial Research Institute, Osaka, AIST
	Government Industrial Research Institute, Kyushu, AIST
(2) Reliability Evaluation Technology	
1) Clarification of Fracture Mechanisms	National Institute for Research in Inorganic Materials, STA
2) Assurance Test Technology	Fine Ceramics Technical Research Association
3) Non-Destructive Test Technology	Fine Ceramics Technical Research Association
3. Application Technology	
(1) Design Technology	
1) Structural Analysis/Design Standards	Fine Ceramics Technical Research Association
2) Fracture Factor Analysis	Fine Ceramics Technical Research Association
(2) Model Evaluation Technology	
1) High-Strength Model	Fine Ceramics Technical Research Association
2) High-Corrosion Resistance Model	Fine Ceramics Technical Research Association
3) High-Precision Wear Resistant Model	Fine Ceramics Technical Research Association

1. Objectives and Outlines, First Phase

As mentioned in the preface, the first phase was for three fiscal years beginning in FY81 and ending in FY83. The R&D efforts during this period were focused on solving the question, whether or not it is possible to achieve the unprecedentedly high Weibull constant values set forth as the targets for various categories of ceramics even with test pieces, and on satisfying the curiosity as to how ceramics' strength would change after exposure to a high-temperature atmospheric environment for many hours. It was impossible to prove improved physical properties of raw materials based on research results directly with ceramic materials

due to the parallel development. However, the central issue in the raw material powder synthesis technology was the firm establishment of fundamental technological elements for producing powders that would be of high purity and microparticles with controlled particle size distribution and crystalline phases, because the production of such powders was the starting point for subsequently clarifying the interrelationships between raw materials and ceramics. For this specific purpose, although it was not clearly indicated in the basic plan document, the tentative target values were established for raw material powders, as shown in Table 1, and these values were used as the standards for the phase-end evaluations.

Table 1. Target Property Values for Synthetic Raw Material Powders

Raw Material	Process	Real Density (g/cm ³)	Specific Surface Area (m ² /g)	Average Particle Diameter (mm)	α -Phase Content (percent)	Purity* (percent)	Total Metal Impurities (ppm)	Fe (ppm)	Ca (ppm)	Al (ppm)	Total Oxygen (percent)
Silicon Nitride	Silicon reduction	>3.11	>6	equal to or less than 0.8	>95	>97.5	<500	<100	<100	<100	—
	Metallic silicon nitrification	>3.11	>20	equal to or less than 0.5	>90	>97.5	<750	<120	<100	<300	—
	Silicon halide nitrification	>3.10	>6	equal to or less than 0.9	>90	>98	<300	<30	<20	<100	—
Silicon Carbide	Solid phase synthesis	>3.10	>15	equal to or less than 0.6	—	>98	<300	<50	<70	<20	<0.5

*Definition of purity: 100—(total impurities + Si in free silica + free Si + C + O + Cl) for silicon nitride; 100—(total impurities + free silica + free Si + free C) for silicon carbide

In order to synthesize the above-mentioned raw material powders, which are the starting point of a ceramic production process, several methods, including the solid-phase as well as gas-phase syntheses, for silicon nitride and silicon carbide were studied simultaneously. The methods were screened, considering test results on synthesized powders' physical properties and the economic aspects of commercial applications. As a result, both silicon nitride powder, made by the silica reduction method, the metallic silicon nitrification method or the silicon tetrachloride-ammonia gas phase reaction method, and silicon carbide powder made by the elemental reaction between metallic silicon and a carbonous material were judged satisfactory. The powders met the tentative target values and were regarded the world's best-quality powder raw materials. During the course of these studies, a considerable number of incidental achievements in technology and cost improvement were also made with respect to unit processes including starting material refining and material handling.

As far as the forming and sintering technology is concerned, the approaches to develop three categories of materials, i.e., high-strength, highly corrosion resistant and high-precision wear resistant materials, from each silicon nitride and silicon carbide were studied by private corporations, which divided the studies among themselves. The process development for each material category for each ceramic material was begun by selecting a forming and sintering method that was considered optimal from the standpoint of the production of an ultimate model shape and the manifestation of desired characteristics in each model. However, because time did not allow the application of raw material synthesis experiment results, whatever raw materials on hand were used for the process development, and their selection was made by the researchers-in-charge, respectively. Common approaches used in these studies were

mainly to study the extraction of effective factors and the process control, to prevent non-uniformities or flaws from being introduced into the material structure in order to attain high Weibull constant values, and to positively prove the findings. Measurements were also made of the properties of in-process working materials for the purpose of theoretically optimizing process conditions. The strength of obtained ceramics was examined as follows: at least five 100 mm square x 10 mm thick ceramic blocks were prepared, from which more than 30 test pieces were cut; the test pieces were used to determine flexural strength according to the JIS specifications; Weibull constant values were obtained via the least square method from the strength value distribution; and tensile strength values were obtained by calculation using the Weibull constant values from the flexural strength values. Most of the physical property values, including the strength values, for ceramics to be evaluated were double-checked by both corporations in charge of the development and GIRI, Nagoya, by testing halves of the same test pieces at two locations. As a result, the ceramic materials, that satisfy the target strength values for test pieces and the target property values for all the categories, were successfully produced. Although, strictly speaking, the target Weibull constant values have never been realized in some cases, these failures were considered within the range of fluctuations. Thus, the Assessment Committee judged that the R&D objectives of the first phase were attained on the basis of the research activities that had produced the successful results.

In order to test the performance of the above-developed ceramics in a model form in the second phase or later, examination was begun in the second phase on the specifications for the models in various categories. Discussions on the significance and methodology of the tests were held between the side in charge of production process technology and the side in charge of evaluation-application technology. It was concluded early in the

second phase that the high-speed revolution test (spin test) at both room temperature and a high temperature was the most suitable method to test high-strength ceramics. Thus, efforts were made to develop high-speed photographic technology to analyze test results. Eventually the trigger method, that could catch the moments of fracture in the order of μsec , was successfully developed.

In FY87, the last fiscal year of the second phase, the fabrication technology research work was first started by private corporation consignees, because the fabrication-bonding technology was the final stage of the production process technology. The work began with preliminary tests for developing composite fabrication technology and high-precision fabrication devices in order to improve the precision, speed and cost of ceramic fabrication. As for the evaluation and application technologies, after preliminary survey research, the systematic strength measurement experiments with standard silicon nitride samples and strength data collection began in the middle of the second phase to clearly define the ceramics fracture criteria from which the design standards were to be deduced in the second phase.

Although there were no definite phase division for the research topics assigned to the government research organizations, during the period corresponding to the first phase, GIRI, Nagoya, and GIRI, Osaka, emphasized the measurement of physical properties of raw material powders and ceramics, or analytical technology research. At the end of the first phase, the results of these efforts were used for evaluation of the developed raw materials and ceramic materials. Other government institutions carried out basic research work, some starting in FY82, the mid fiscal year of the first phase, as described in the respective reports in this book.

1. Objectives and Outlines, Second Phase

In the second phase, FY84 through FY87, the standard for judging its overall results was focused on the successful attainment of the respective target physical property values established in the basic plan for simple-shaped models (primary models) and for ceramic materials. The models were to be made from the three categories of developed ceramic materials of silicon nitride and silicon carbide by going through the entire production process from the raw materials to the finishing fabrication processes. Also in the second phase, R&D results concerning each individual elemental technology were evaluated for all the research topics. The following test methods were proposed to check the improvement status of the above-mentioned physical property values through comprehensive performance tests with the models: the high-speed revolution test with disc-shaped models, designed for almost equal stress loading, to be used for the high-strength ceramic materials; the fracture test by variable thermal stress with cylindrical models for the highly corrosion-resistant ceramic materials; and a set of three tests consisting of the air bearing, friction wear and abrasion wear tests with suitably shaped models, respectively. The detail

model specifications and test conditions were decided after heated discussion between the group in charge of production process and the group in charge of evaluation and application, and in the presence of government institution representatives. Subsequently, both sides simultaneously worked on model preparation and on test device production and function tests for the purpose of upgrading the production process technology. By the end of the second phase, the corporations in charge of processes submitted models to the corporation in charge of testing, and the tests were carried out separately for each material category. Test details are described in respective reports. Although the tests confirmed that all the models showed satisfactory performance to attain the target values, the Weibull constants for the strength values obtained with test pieces cut out from the silicon carbide models were significantly below the target values except for one category. These results proved the difficulty in homogeneously forming and sintering ceramics with a shape. This fact gave clues to not only the methods to evaluate research results, but also the design and use methods for parts to be actually used during and after the third phase. The raw material powders for preparing models were to be selected for their suitability, in principle, from all the powder materials that had been developed in the first phase of this project and had been since studied in interim-scale mass-production experiments. Those corporations responsible for raw material synthesis pursued R&D on the synthesis, gradually increasing their synthesis capacities, and supplied research quantities of the resulting powder raw materials to the sintering technology-developing corporations. At any time during this period, the specifications for these powders were amended to meet the users' demands by mutual agreement between the synthesizer and the evaluator. Also, during this period, joint industry-government studies as well as subconsigned cooperative research work with the participation of universities were carried out in order to systematically clarify the correlations among raw material powders' characteristics and sinterabilities and ceramics' characteristics.

Parallel with the above-described mainstream research activities, many side R&D projects were carried out to improve the sophistication of individual technologies within each of the three technology divisions composing the main project, and noteworthy results were obtained for the side projects. Details of these results are given in the reports under the respective topics. To pick a few examples, the anti-oxidation property (in terms of the weight gain after high-temperature oxidation) of highly corrosion-resistant silicon nitride reached the world's highest level by the post-sintering technique; in the area of fabrication technology, not only the technology itself was improved, but also the compound fabrication principle, that aimed at improving fabrication efficiency and cost by compounding the machine cutting process, was sought and selected, and the successful development of a high-rigidity precision cutting device was instrumental in realizing the world's highest machining efficiency and machining ratio. The highest value was also obtained for

adhesive strength between silicon nitride and steel. For the non-destructive test methods for ceramics, techniques that had previously never been explored were mainly examined. Thus, first the PAS (photo-acoustic spectrography) method was found to be effective, in principle, for detecting surface flaws, and subsequently, the XCT (X-ray computer tomography) method was studied to check its applicability. In the area of application technology, the reliability in ceramics' physical property measurements was verified, because it was an important part of the effects of ceramic application. After the verification, an enormous amount of measurements were made on both instantaneous and time-dependent ceramic fracture behaviors against all kinds of simple and complex stresses, and on the basis of the results, which were analyzed and accumulated, the design standards for ceramic parts were proposed for the first time in the world. In addition, a high-precision computing system was developed for use in the analysis of stresses and reliabilities needed for design, and during the second phase, the system became applicable for two-dimensional designs. A new analytical method, that could be applied when there was a discrepancy in stress distributions between proof tests and service conditions; and an evaluation method, based on data obtained with an insufficient number of samples, were newly proposed in the area of assurance tests and life prediction methods for ceramic parts.

As shown in Table 2 of the preface, the main parts of the ceramic evaluation and measurement technology development were charged to government research organizations and continued from the first phase. Details of the activities are described in the fourth chapter of this book under the title of "Basic Research."

Based on all the above-described R&D achievements, the Assessment Committee again recognized that the R&D targets established for the second phase had been successfully attained.

Starting in the late first phase, research concerning common and basic problems was consigned to national universities at one topic per institution and for two years. By the end of the second phase, the following three were completed:

"Basic Research concerning Sintering of Silicon Nitrides," Professor Akio Kato, Faculty of Engineering, Kyushu University

"Basic Research concerning Physical Properties and Formabilities of Powders," Professor Masafumi Arakawa, Faculty of Engineering, Kyoto Institute of Engineering

"Effects of Machining on Bending Strength of Various Silicon Carbide Ceramics," Assistant Professor Yotaro Matsuo, Faculty of Engineering, Tokyo Institute of Engineering

For details of the above topics, readers are asked to refer to the respective dissertations by these professors who led these studies.

Expansion of Research Topics

Subsequent to the inauguration of this R&D project by the consignment of the general account budget of AIST, in FY86, the electrical power resource development promotion special account budget was allocated to a portion of the project, and further in FY87, with the exception of one research topic under the jurisdiction of the National Institute for Research in Inorganic Materials, STA, all other topics came under the above special account budget. With this account change, the characteristics of the R&D project was somewhat modified from the original direction of pure material science and technology-orientation to the more objective-oriented type. At the same time, the project title was renamed as follows: "the coal gasification ceramics turbine elemental technology development" under the responsibility of the Fine Ceramics Technical Research Association, and "the coal gasification ceramics turbine elemental evaluation method development" under the responsibility of the government research organizations under the jurisdiction of AIST. Furthermore, the ultimate target for proving developed technologies had to be focused on various elemental technologies involved in developing and applying gas turbine ceramic parts for the core devices in "the coal gasification cogeneration system," which is one of the most promising high-efficiency power generation methods then and in the future. However, the previous research philosophy and methodology toward key technology development were still maintained to ultimately carry out the verification of material technology research results with realistic objects. Therefore, it would be essential to understand and apply the systematic knowledge on all material technologies that could offer the most effective technical guidelines to solve various problems concerning the production and usage of the above-mentioned gas turbine ceramic parts. According to the basic plan, the third phase was the period to realize the expected performance with complex-shaped models. However, it was suspected that the performance range of the developed research ceramic materials with respect to their adaptation to realistic environments were inadequate for achieving the above-mentioned objectives. Thus, a study was conducted to improve the materials' performance, and at the same time, in the second half of FY87, i.e., essentially in the beginning of the third phase, the following new research topics were added: the development of toughness reinforcing materials and surface strengthening technology, the development of instrumentation technology for mechanical shocks, and the establishment of design standards. Therefore, as mentioned in the preface, the research period was extended by two years, the basic plan diagram was revised, and, furthermore, the key target performance items were amended as shown in Table 1 below. In an attempt to speedily and

simultaneously examine more than one technical possibility, the R&D work on toughness reinforcing materials and surface strengthening technology was divided into five Table 2- listed subtopics with 10 approaches, all of which are now being pursued on the assumption that the selection of an optimal approach be made as soon as

possible. Further studies are expected in case these new materials require new fabrication technology and evaluation technologies, including non-destructive tests, physical property measurements, and thermal fatigue properties, which are different from the previous ones in principle and viewpoint involved.

Table 1. Target Performances for the Third Phase

Ceramic Material Field	Target Values
Materials targeted for use at temperatures higher than 1,400°C	(1) Fast fracture strength: to satisfy performance values listed below in an atmospheric environment at temperatures higher than 1,400°C
	1) Minimum guaranteed strength: more than 400 MPa
	2) Rejection rate: less than 20 percent
	3) Weibull constant (reference value): more than 20
	(2) Delayed fracture strength: to satisfy performance values listed below in 10,000 hour-equivalent creep tests in an atmospheric environment at temperatures higher than 1,400°C
	Creep strength: more than 250 MPa
	(3) Oxidation, corrosion and wear resistance: to show neither significant strength change nor quality deterioration after exposed to a coal ash-containing combustion gas flow for more than 200 hours at temperatures higher than 1,400°C
	(4) Fracture toughness value: if a material is likely to satisfy all the above conditions, it must satisfy the following performance value in a room-temperature test:
	Fracture toughness value: more than 8 MPam ^{1/2}
Materials targeted for use at temperatures higher than 1,250°C	(1) Fast fracture strength: to satisfy performance values listed below in an atmospheric environment at temperatures higher than 1,250°C
	1) Minimum guaranteed strength: more than 600 MPa
	2) Rejection rate: less than 20 percent
	3) Weibull constant (reference value): more than 20
	(2) Delayed fracture strength: to satisfy performance values listed below in 10,000 hour-equivalent creep tests in an atmospheric environment at temperatures higher than 1,250°C
	Creep strength: more than 250 MPa
	(3) Oxidation, corrosion and wear resistance: to show neither significant strength change nor quality deterioration after exposed to a coal ash-containing combustion gas flow for more than 200 hours at temperatures higher than 1,250°C
	(4) Fracture toughness value: if a material is likely to satisfy all the above conditions, it must satisfy the following performance value in a room-temperature test:
	Fracture toughness value: more than 15 MPam ^{1/2}

Table 2. New Material Technology Research Subtopics and Number of Approaches

(1) Toughness Improving Materials	
1) Particle-dispersion type high-toughness materials	Three approaches
2) Fiber-reinforcement type high-toughness materials	Four approaches
3) Crystal growth control type high-toughness materials	One approach
(2) Surface Strengthening Technologies	
1) Strengthening technology by surface modification	One approach
2) Strengthening technology by surface coating	One approach

Prospects and Expectations for the Final Phase

The inauguration of the "Fine Ceramics R&D Project" under the next generation's industrial key technology R&D system attracted interests of the advanced nations worldwide, and in the first phase, frequent inquiries were made by foreign countries for status survey. In particular, the 1982 visit by French government representatives and the 1983 visit by the U.S. representatives

from the National Academy of Sciences merit special mention, because these visits made impacts on the governmental guidelines concerning new material development in the West. Since the early 1970's, the United States had carried out several research projects under the government's initiative to develop ceramic automobile part materials, and was concluding the so-called AGT project when the next generation system was established.

Also in West Germany, the development of automobile gas turbines based on the cooperation agreement with the United States had been under way with the participation of the industry, government and universities under the management of the Aerospace Research Institute and with the support from the Science and Technology Department. However, in both the United States and West Germany, automobile manufacturers had happened to be the project consignees, whose main efforts had been in the system development with the material development placed in a subordinate position. However, in the United States in the very same year when the visit took place, the new national research project, the ceramic technology project for advanced heat engines, was initiated. Several years after the visits, since both European and the U.S. previously initiated gas turbine development projects were to be terminated without gaining advantages either for future research or in realistic cost, the projects were replaced by the new ones attaching importance to the materials. These events prove nothing but the importance and difficult nature of the ceramic material technologies.

The material technology-oriented posture will always be maintained in the next generation's research project. The essential intention of the material technology development is not to develop technologies of sectional assembly in order to produce specific parts, but is rather to necessarily arrive at technologies, that will permit the production of highly reliable products, as a result of the constant reminding of the elucidation of the total picture of technologies, despite wastes of time and labor incurred on the way. This is the only possible way to discover currently unknown materials and unknown uses.

The problems confronted in the third phase include the existing material improvement and new material development and the selection of model specifications and test methods that are suitable for proving the attainment of goals. Heavily demanded, in addition to the previous research topics, will be the one-step deeper study on the relationships between the factors concerning raw material powders and additives, the material performance improvement and the material toughness improvement effects, as well as the pursuit in the wide-ranging experiments and surveys on the corrosion resistance aspects of ceramics, the development of comprehensive application technology of non-destructive tests and the development of product life prediction methods.

In the next generation's research project, a considerable range of ceramic material technologies is covered. However, the scope of related technologies is even broader. The constant communications and coordination with the entire ceramic technology community, which is rapidly advancing at both the domestic and international fronts, are indispensable for obtaining truly significant results from the next generation's research project. Thus, it would

be fortuitous if this book could play the role of the liaison. Because of this R&D project, the attitude of conversation and cooperation was born and the exchange of information, which was recognized as the common property, became fluid among the participating corporations, which had once been fierce competitors, contributing to the improved quality of results. It is hoped that further advancement of the ceramic material technology based on such cooperation will be continued not only in all over Japan but also in all over the world.

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